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## High Temperature Fatigue of Nickel-based Superalloys during High Frequency Testing

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### Abstract

Microstructural features acting as stress raisers lead to localised and inhomogeneously distributed irreversible deformation in the very high cycle fatigue (VHCF) regime although load amplitudes are well below the classical fatigue limit. Hence, changes in the microstructure due to thermally activated processes can result in an overall change of damage mechanism compared to the fatigue behaviour at room temperature. The influence of isothermal testing at elevated temperatures up to 800°C during high frequency testing was studied with regard to the VHCF behaviour of the nickel-based superalloys Nimonic 75 and 80A. This material combination allowed a distinction between the influence of the stability of the initial precipitation condition (precipitation-free, peak-aged, overaged) on the one hand and the formation of oxide layers on the other hand. High frequency tests were accompanied by extensive metallographic analysis by means of high resolution scanning and transmission electron microscopy. Early failure was primarily ascribed to the formation of microcracks in the emerging oxide layers. In this respect, the relevance of high frequency testing regarding the true fatigue behaviour, which in the case of thermally activated microstructural changes implies a likely time dependence of damage mechanisms, will be critically reviewed.

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### 1. Introduction

The combination of cyclic loading together with high temperature environment makes high demands on the strength behaviour of materials, all the more so when a lifetime beyond the classical fatigue limit is required. Nickel-based alloys as material for turbine blades or discs face such challenges as their expected in-service loading conditions include high frequency loading at low amplitude levels in combination with exposure to elevated temperatures [1,2]. Due to the globally purely elastic amplitudes in the very high cycle fatigue (VHCF) regime, local stress raisers such as micronotches, and the dislocation-precipitation interaction dominate the damage evolution and the likeliness of crack initiation and growth under the given circumstances [3,4]. However, extended cyclic loading at elevated temperatures also introduces the question of indirect

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frequency effects and hence the occurrence of creep phenomena and high temperature oxidation [5,6]. In the study presented the polycrystalline wrought alloys Nimonic 75 and Nimonic 80A were analysed. Another nickel-based superalloy, René 88DT was studied in the VHCF regime at 593°C but did not exhibit any corrosion or creep effects while cracks initiated at large grains with  $\Sigma 3$ -twin boundaries and high Schmid factor [7]. In principle, fatigue crack in Ni-based superalloys can emanate from planar slip bands, grain boundaries, pores and inclusions (in cast and powder-metallurgy alloys) [8]. With an increasing influence of microstructural inhomogeneities on the fatigue behaviour in the VHCF regime, the analysis of competing failure modes and the development of an appropriate fatigue life prediction model get all the more challenging.

## 2. Material and experimental setup

Nimonic 80A was tested in the peak-aged and the overaged conditions, both defined on the basis of Vickers hardness tests and an analysis of the geometry, size and distribution of  $\gamma'$  precipitates by means of transmission electron microscopy (TEM). Nimonic 75 was solely coarse-grained and annealed in order to adapt the grain size to that of Nimonic 80A and to avert any grain size effect on the dislocation slip behaviour. The single-phase alloy Nimonic 75 represents the behaviour of the  $\gamma$  matrix of Nimonic 80A. The chemical composition matches almost perfectly except for the iron concentration. Details on the heat-treatment, the chemical composition and the static strength data for both alloys can be found elsewhere [9].

Fatigue tests were executed with hourglass-shaped specimens with a mechanically ground and electrochemically polished surface. Test frequencies varied according to the test systems used:  $\sim 130$  Hz for a resonance electromechanical device, 760 Hz for a servohydraulic high-frequency system and  $\sim 19.5$  kHz for an ultrasonic fatigue test system which was solely used for reference tests at room temperature. A sample was denominated as run-out when at a given number of cycles optical inspection did not show any signs of crack initiation at the surface. During testing at room temperature the samples were cooled by means of compressed air and were cycled in pulse/pause sequences for ultrasonic testing. For high temperature fatigue testing, an induction heating system was applied. All fatigue tests were carried out with a stress ratio  $R = -1$ . TEM samples were cut from the highest stressed region of the fatigued samples. Details on the steps applied in TEM sample preparation can be found in [5].

## 3. Results and discussion

Fatigue life (as is shown in the S-N-curves given in Fig. 1) at room temperature in the VHCF regime of Nimonic 80 A and Nimonic 75 is dominated by the dislocation-precipitation interaction. A more homogeneous dislocation slip arrangement in the overaged condition leads to an enhancement of the fatigue strength compared to the peak-aged material. The precipitation free Nimonic 75 shows the lowest cyclic strength at very high number of loading cycles, as would have been expected. Isothermal fatigue tests at 800°C reveal a drastic decrease of cyclic strength for all precipitation conditions, while at intermediate temperatures (400°C and 600°C) hardly any change in fatigue life can be observed when compared with room temperature results. However, a change in dislocation mobility is already evident at these temperatures. At room temperature dislocation slip bands either form in single bands throughout the grain or pile-up at cuboidal overaged precipitates (Fig. 2a). Between 400°C and 600°C, a change from mainly planar slip character to a more wavy slip mode, including Orowan bypassing, can be observed both for peak-aged and overaged Nimonic 80A (Fig. 2b), resulting in a slightly more homogeneous formation of slip bands. Yet, slip activities induced by VHCF loading are still limited to isolated grains. Orowan loops and diffusion-controlled climbing processes can be confirmed for fatigued samples at 800°C (Fig. 2c). However, fatigue life at this temperature level is no longer only defined by the dislocation/particle interaction. Cycling up to a number of loading cycles  $N > 10^7$  leads to test periods surpassing a time interval of 3.6 hours for test frequency of  $f = 760$  Hz and 21 hours for a test frequency of  $f = 130$  Hz and for  $N > 10^8$  beyond 1.5 and 8.5 days, respectively. Under the given test conditions, particle coarsening, recovery processes and the formation of an oxide layer have to be taken into consideration. Furthermore, the possibility of damage phenomena similar to a creep/fatigue interaction at test periods comprising several days at a temperature of 800°C has to be discussed.

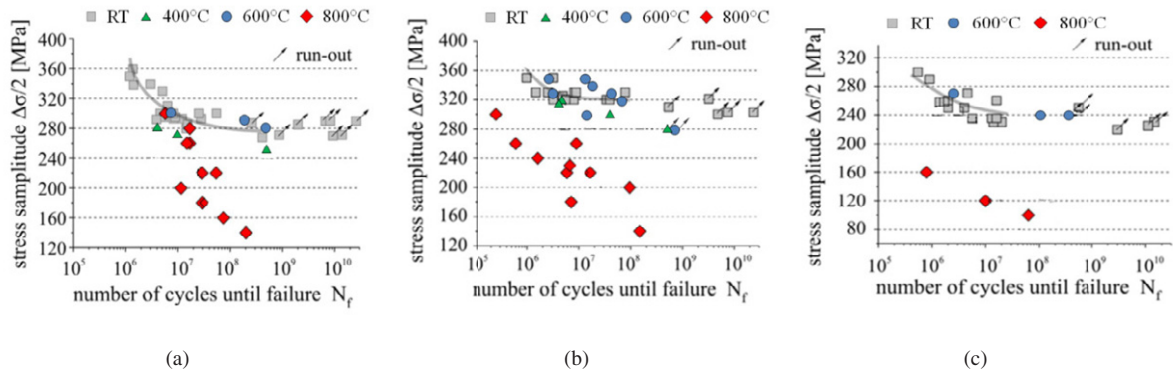


Fig. 1. S-N-curves for peak-aged Nimonic 80A (a), overaged Nimonic 80A (b) and Nimonic 75 (c) at room temperature and at elevated temperatures [5,9].

With regard to the influence of particle coarsening (Fig. 2d) and recovery processes it can be stated, that both processes could be observed during fatigue testing at intermediate temperatures and the slightly superior cyclic strength for the peak-aged condition at 600°C compared to the results at 400°C in the VHCF regime can be explained by the coarsened particles, hence showing a more effective barrier function against the formation of single dislocation slip bands (comparable to the effect of dislocation/particle interaction in the overaged condition). However, comparing the effect of particle coarsening and the change of slip mode due to thermally activated climbing, the latter one can be identified as the dominating effect. Since for the precipitation-free condition, represented by Nimonic 75, the influence of a change from planar to pronounced wavy slip mode dominates the fatigue behaviour at 600°C, its fatigue strength is not affected by this (application relevant) temperature.

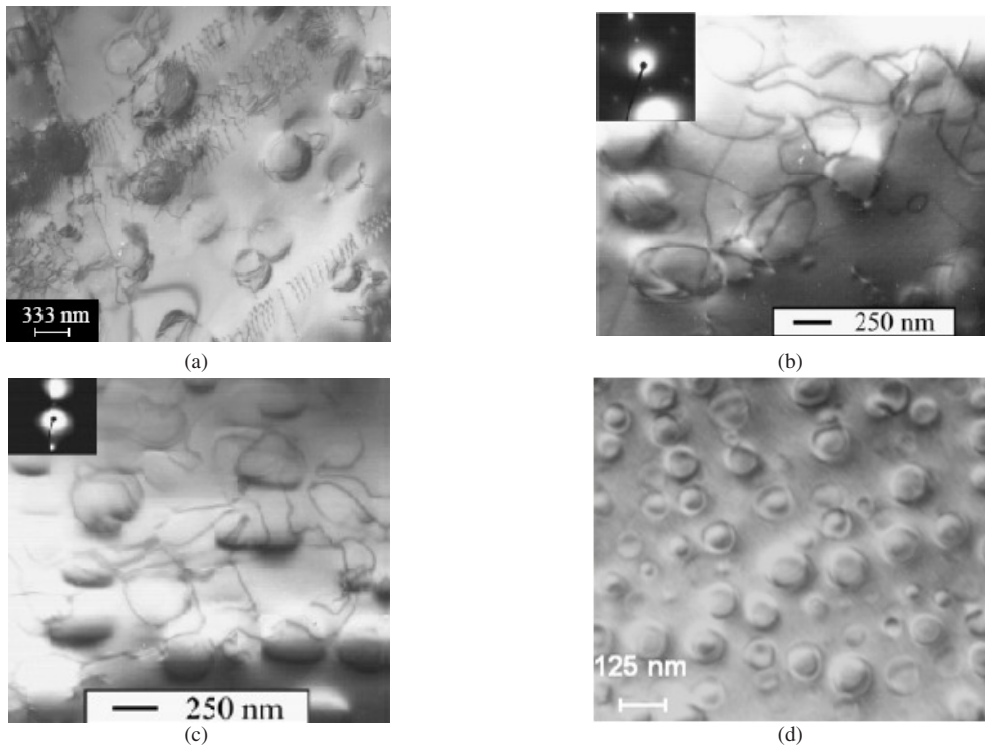


Fig. 2. Dislocation/particle interaction during fatigue a) at room temperature [4] b) at 600°C and c) at 800°C for the overaged and d) particle coarsening at 800°C for the peak-aged condition [11].

The long overall testing time results in the formation of oxide layers, which at intermediate temperatures are still very thin (less than 1  $\mu\text{m}$ , Fig. 3a), while cycling at 800°C evokes the formation of a pronounced brittle oxide layer (Fig. 3b) with an increased notch sensitivity. In addition, embrittlement at the grain boundaries is caused by the formation of chromium carbides in turn promoting intergranular oxidation. An earlier crack initiation is the consequence resulting in a pronounced decrease in fatigue life at 800°C. The formation of a  $\gamma'$  film coating the carbides led to a further weakening of the grain boundaries. The effect of the brittle oxide layer together with a localized notch stress at the tip of slip bands and the weakened grain boundaries is confirmed by the intercrystalline manner of the fracture surface (Fig. 3c), leading to the assumption of a likely creep/fatigue interaction as dominating cause for crack initiation. In comparison, fatigue testing at 600°C produced a fracture surface with both transgranular as well as intergranular fractions (Fig. 3d). However, grain boundary carbides, as were observed in the specimens tested, usually improve the creep properties and prevent grain boundary sliding [10]. Moreover, whether a true creep loading condition has been introduced by the high temperature and long test period is yet to be confirmed, as all fatigue tests were executed at a stress ratio of  $R = -1$ . A comparison of the results presented with VHCF tests at a positive  $R$  ratio combined with dwell times at tensile loading would contribute to a clarification of the cumulative effects of fatigue, creep deformation and oxidation for the two nickel-based superalloys during isothermal VHCF loading. As creep causes cavity formation in the oxide layer or at oxide-matrix interfaces [6], which in turn might influence the fatigue crack growth behaviour of microcracks, future investigations' aim should be to separate these effects.

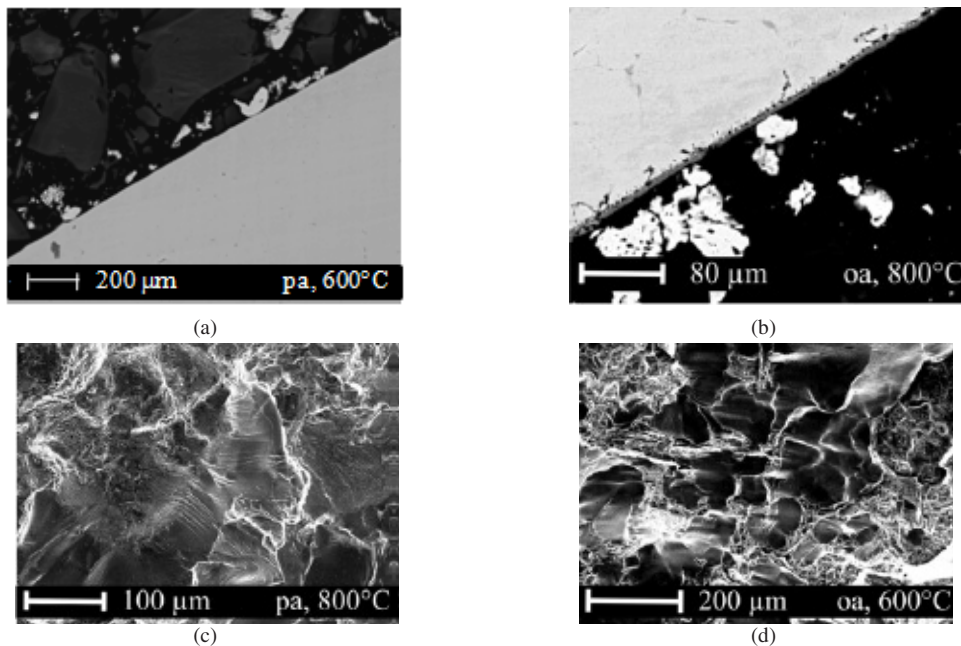


Fig. 3. Formation of an oxide layer during fatigue testing at (a) 600°C and (b) 800°C and corresponding fracture surfaces (c) for 800°C and (d) 600°C. [9].

#### 4. Conclusion

The VHCF behaviour of the precipitation-hardened nickel-based superalloy Nimonic 80A and the precipitation-free alloy Nimonic 75 is dominated by the dislocation structure in isolated grains (homogeneous versus inhomogeneous distribution), which in the case of Nimonic 80A strongly depends on the dislocation/particle interaction at room temperature with slightly superior fatigue strength for the overaged condition. Fatigue properties in the high-temperature range are revealing a combination of different time and

temperature dependent effects. Up to intermediate temperatures (400°C and 600°C) a change from planar to wavy slip mode results in a more homogeneous distribution of slip bands in isolated grains, hence, fatigue life is comparable to room temperature fatigue life regardless of the precipitation condition. In contrast, isothermal cyclic deformation at 800°C led to a drastic decline in cyclic strength, with number of cycles no longer extending into the VHCF range. The underlying failure mechanisms are discussed in the context of oxidation processes and creep/fatigue interaction. Microcrack formation at the brittle oxide layer combined with intergranular crack propagation due to weakened grain boundaries (due to intergranular oxidation and carbide segregation) was identified as predominant reason for the early fatigue failure. However, future investigations pursuing a separation of creep and oxidation effects might further elucidate the complex failure mechanisms under combined VHCF and high temperature testing at different test frequencies.

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